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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE APPLICATION OF: GROUP: 1793

Hans-Detlef LUGINSLAND, et al.

SERIAL NO: 10/542,850 EXAMINER: PARVINI

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FOR: SPECIALLY PRECIPITATED SILICIC ACIDS FOR RUBBER APPLICATIONS

DECLARATION UNDER 37 C.F.R. § 1.132

COMMISSIONER FOR PATENTS
ALEXANDRIA, VIRGINIA 22313

Sir:

Now comes André Wehmeier who deposes and states that:

1. I am a graduate of FH Münster and received my diploma degree in the year 1998.
2. I have been employed by Evonik Degussa GmbH for 10 years as a chemical graduated engineer in the field of product development silica.
3. The following experiments were carried out by me or under my direct supervision and control.

Enclosed is a comparison experiment in which the properties of the silica according to Example 3 of US 5,846,506 are compared with those of the silica III of the Examples of the present invention (see page 35, starting at line 30 of the specification).

The precipitated silicas of the present invention have clearly improved processing properties because of the higher ratio of Sears number to BET. In other words, using the silicas of the present invention leads to drastically shorter vulcanization times, greater vulcanization rates and lower Mooney viscosities.

The enclosed comparison example therefore confirms the statements starting at page 16, line 28 of the specification, according to which the increased number of silanol groups per m^2 of surface area leads to a faster and more efficient hydrophobation (the bonding of the

coupling agent (silane) to the silica surface). As a consequence of this additionally the vulcanizate data are strongly improved regarding reinforcement, abrasion resistance and hysteresis loss values.

The comparison example clearly demonstrates the relevance of the ratio of Sears number to BET and also shows that this parameter leads to a considerable improvement in performance of the silica of the present invention compared with the silica of US 5,846,506.

General Standard Operating Procedure (SOP):

The silica of the present invention was tested in a typical passenger car tire tread compound. The formula used for the rubber mixtures is presented in the following Table 1, in which the unit "phr" means parts by weight relative to 100 parts of the raw rubber used.

Substance	phr	Article designation	Company
1st step	**Basic mixing**		
Buna VSL 5025-1	96.0	S-SBR; oil-extended	Lanxess Europe GmbH & Co. KG; 51369 Leverkusen; Germany
Buna CB 24	30.0	cis-1,4-BR	Lanxess Europe GmbH & Co. KG; 51369 Leverkusen; Germany
Silica	80.0		
X 50-S	12.8	Si 69 (bis(3-triethoxysilylpropyl)tetrasulfane) / carbon black of type N 330: 50% / 50%	Evonik Degussa GmbH; 45128 Essen; Germany
ZnO; RS RAL 844 C	3.0	ZnO	Amsperger Chemikalien GmbH; 50858 Cologne; Germany
EDENOR ST1 GS	2.0	Palmitic-stearic acid; "iodine number 1" stearin	Caldic Deutschland GmbH & Co. KG' 40231 Düsseldorf; Germany
Naftolen ZD	10.0	Aromatic plasticizer oil	Chemetall GmbH; 60487 Frankfurt a. Main; Germany
Vulkanox 4020 / LG	1.5	N-(1,3-Dimethylbutyl)-N'-phenyl-p-phenylenediamine (6PPD)	Rhein Chemie Rheinau GmbH; 68219 Mannheim Rheinau; Germany
Protektor G 3108	1.0	Mixture of refined hydrocarbon waxes	Paramelt BV; 706875 Paramelt BV; NL 1704 RJ Heerhugowaard; The Netherlands
2nd step	**Remill step**		
Step 1 batch			
3rd step	**Finish mixing**		
Step 2 batch			
Vulkacit D	2.0	N,N'-Diphenylguanidine (DPG)	Rhein Chemie Rheinau GmbH; 68219 Mannheim Rheinau; Germany
Vulkacit CZ/EG-C	1.5	N-Cyclohexyl-2-benzothiazole sulfenamide (CBS)	Rhein Chemie Rheinau GmbH; 68219 Mannheim Rheinau; Germany
Perkacit TBZTD	0.2	Tetrabenzylthiuram disulfide (TBzTD)	Flexsys N.V./S.A., Woluwe Garden; B-1932 St. Stevens Woluwe; Belgium
Ground sulfur	1.5	Finely divided sulfur according to Ph Eur, BP	Merck KGaA; 64271 Darmstadt; Germany

Table 1: Formula for a passenger car tire tread compound

The general method for manufacture of rubber mixtures and their vulcanized derivatives is described in the following book: "Rubber Technology Handbook", W. Hofmann, Hanser Verlag 1994. The specific mixing conditions for the various compounds are presented in Table 2.

1st step	Brabender 350 S mixer, filling level 0.73, 70 rpm, chamber temperature 70 °C, friction 1 : 1.11 ramp pressure 5 bar
0.0 to 0.5 minutes	Polymers
0.5 to 1.5 minutes	1/3 silica, X 50-S
1.5 minutes	Clean
1.5 to 2.5 minutes	1/3 silica
2.5 minutes	Clean
2.5 to 3.5 minutes	1/3 silica, ZnO, stearic acid, oil, Vulkanox 4020, Protektor
3.5 minutes	Clean
3.5 to 5.0 minutes	Mix; with speed variation if necessary, in order to reach the dump temperature
5.0 minutes	dump batch (batch temperature 145 °C to 155 °C) and process on an open mill: Cut in and fold over 3 * on left, 3 * on right, Roll up and pass through a tight nip 5 *, through a wide nip 5 * Sheet off
24 hours intermediate storage at room temperature to step 2	
2nd step	Brabender 350 S mixer, filling level 0.71, 80 rpm, chamber temperature 80 °C, friction 1 : 1.11 ramp pressure 5 bar
0.0 to 2.0 minutes	Plasticize batch from step 1
2.0 to 5.0 minutes	Maintain batch temperature at 150 °C by speed variation
5.0 minutes	dump batch (batch temperature 145 °C to 155 °C) and process on an open mill: Cut in and fold over 3 * on left, 3 * on right, Roll up and pass through a tight nip 5 *, through a wide nip 5 * Sheet off
4 hours intermediate storage at room temperature to step 3	
3rd step	Brabender 350 S mixer, filling level 0.69, 50 rpm, chamber temperature 60 °C, friction 1 : 1.11 ramp pressure 5 bar
0.0 to 0.5 minutes	Batch from step 2
0.5 to 2.0 minutes	Constituents of the 3rd step
2.0 minutes	dump batch (batch temperature 90 °C to 110 °C) and process on an open mill:: Cut in and fold over 3 * on left, 3 * on right, Roll up and pass through a tight nip 5 *, through a wide nip 5 * Sheet off in the thickness necessary for

	preparation of the test specimens
12 hours intermediate storage at room temperature until vulcanization of the test specimens	

Table 2: Mixing SOP

Technological rubber testing takes place according to the test methods presented in Table 3.

Physical testing	Standard / Conditions
ML (1+4), 100 °C, 3rd step (MU)	**DIN 53523/3 ISO 667**
Vulcameter test, temperature, 165 °C, strain 0.5 ° **MDR rheometer** M_L (dNm) t 90% (minutes) t 80% - t 20% (minutes)	**DIN 53529/3 ISO 6502**
Tensile test; dumb-bell S 1, 23 °C **(median values from 3 dumb-bells)** Modulus 200% (MPa) Modulus 200% / modulus 50% (--)	**DIN 53504, ISO 37**
Ball rebound 70 °C (%)	**DIN EN ISO 8307,** drop height 500 mm, steel ball, d = 19 mm, 28 g
DIN abrasion, 23 °C, 10 N force (mm^3)	**DIN 53516**
Viscoelastic properties, **50 N preliminary force and 25 N amplitude force, 16 Hz** **Temperature equilibration time 5 minutes, recording of measured values after 30 s of test time** tan δ, 60 °C (--)	**DIN 53513, ISO 2856**

Table 3: Test methods

Table 4 below presents the application-related data of the mixtures compounded and tested according to Tables 1 to 3.

		US 5,846,506 Example 3	Silica III
ML (1+4), 100 °C, 3rd step	MU	90	87
MDR: 165 °C; 0.5 °			
M_L	dNm	3.5	2.9
t 90%	min	9.7	5.2
t 80% - t 20%	min	3.2	1.8
Vulcanization time (165 °C)	min	20	20
Modulus 200%	MPa	8.0	13.2
Modulus 200% / modulus 50%	–	5.0	7.3
DIN abrasion; 10 N	mm^3	77	62
Ball rebound; 70 °C	%	65.8	69.2
Zwick: 16 Hz; 50 N ± 25 N			
tan δ, 60 °C	–	0.134	0.109

Table 4: Results

The compounds containing the silicas according to the present invention have a profile of rubber values superior to that of the US 5,846,506. For example, the properties of the raw mixture include a lower Mooney viscosity, as confirmed by the M_L value in the MDR test. Thus, improved processing behavior is demonstrated. In addition, the vulcanization time 90% and the vulcanization rate t 80% - t 20% are drastically reduced.

These greatly improved raw-mixture properties can be attributed in particular to the higher and therefore better ratio of Sears number to BET surface area, since hereby faster and more effective hydrophobing and thus greater binding capacity of the silane to the silica are possible.

The greatly improved properties of the vulcanized derivative can also be attributed for the most part to this analytical characteristic. For example, the improved binding of the silicas leads to better reinforcement of the vulcanized derivative in the tension test, as seen in the higher value of modulus 200% and the higher modulus 200% / 50% reinforcement factor. As a consequence, the DIN abrasion is improved by more than 18%. Surprisingly, the hysteresis

behavior, which correlates directly with the rolling resistance of a tire finished with this running-surface compound, can also be improved simultaneously by more than 18% with the inventive silicas (see tan δ, 60 °C). This improvement is also confirmed by the ball rebound value, 70 °C.

The precipitated silicas of the present invention have clearly improved processing properties because of the higher ratio of Sears number to BET. In other words, using the silicas of the present invention leads to drastically shorter vulcanization times, greater vulcanization rates and lower Mooney viscosities. As a consequence of this additionally the vulcanizate data are strongly improved regarding reinforcement, abrasion resistance and hysteresis loss values.

The enclosed comparison example therefore confirms the statements starting at page 16, line 28 of the specification, according to which the increased number of silanol groups per m^2 of surface area leads to improved and better binding of the coupling agent (silane).

The comparison example clearly demonstrates the relevance of the ratio of Sears number to BET and also shows that this parameter leads to a considerable improvement in performance of the silica of the present invention compared with the silica of US 5,846,506.

4. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

5. Further deponent saith not.

Signature

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